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MEMORANDUM

DATE: March 28, 2011

TO: Renee Nordeen, Project Manager, Ecology and Environment, Inc., Seattle, WA

FROM: Mark Woodke, START QA Chemist, Ecology and Environment, Inc., Seattle, WA

SUBJ: TDD: 10-11-0007 Example adjusted CRQL calculation for the Bremerton Gasworks TBA

Example adjusted Contract Required Quantitation Limit (CRQL) calculations for soil samples collected in conjunction with the Bremerton Gasworks Targeted Brownfields Assessment conducted by Ecology and Environment, Inc., in 2008 are provided below. Actual adjusted CRQLs are provided at the end of this memo.

SOIL SAMPLES

The detection limits are derived from the contract required quantitation limits (CRQLs; attached), % solids sample weights, and dilution factors (all on the Form I's), and contract modifications (attached).

CLP Lab Soil/Sediment Inorganics Example Adjusted CRQL Calculation - Method ILM05.4

Adjusted CRQL = Contract CRQL x 1/% solids

Cobalt in sample MJ8K76

Adjusted CRQL = $5.0 \text{ mg/kg} \times 1/0.722 = 6.9 \text{ mg/kg}$

CLP Lab Soil/Sediment Organics Formula

SVOCs

Adjusted CRQL = Contract CRQL x Contract Sample Weight (30 grams)/Actual Sample Weight x Volume of Concentrated Extract/Concentrated Extract Volume (same if GPC is used, so they cancel out) x Dilution Factor/(1/% solids (decimal))

Example Calculation for sample J8K78

Acenaphthene - Method SOM01.2 (SVOC)

The Contract CRQL was modified for this project from $170 \text{ } \mu\text{g/kg}$ to $68 \text{ } \mu\text{g/kg}$

$68 \text{ } \mu\text{g/kg} \times 30 \text{ grams}/100.3 \text{ grams} \times 1/0.82 = 24 \text{ } \mu\text{g/kg}$.

VOCs

Adjusted CRQL = Contract CRQL x Contract Sample Weight (5 grams)/Actual Sample Weight x 1/% solids (decimal)

Example Calculation for sample J8K74

o-Xylene – Method SOM01.2 (VOC)

The Contract CRQL was modified for this project from $5 \text{ } \mu\text{g/kg}$ to $1 \text{ } \mu\text{g/kg}$

$1 \text{ } \mu\text{g/kg} \times 5 \text{ grams}/4.6 \text{ grams} \times 1/0.82 = 1.3 \text{ } \mu\text{g/kg}$.

Adjusted CRQLs for the EPA Targeted Brownfields Assessment

Sample Identification	Hazardous Substance	Adjusted CRQLs
J8K78 (08204462; WN05SD)	Arsenic	1.2 mg/kg
	2-Methylnaphthalene	24 µg/kg
	Acenaphthene	24 µg/kg
	Acenaphthylene	24 µg/kg
	Anthracene	24 µg/kg
	Benzo(a)anthracene	24 µg/kg
	Benzo(a)pyrene	24 µg/kg
	Benzo(b)fluoranthene	24 µg/kg
	Benzo(g,h,i)perylene	24 µg/kg
	Benzo(k)fluoranthene	24 µg/kg
	Carbazole	24 µg/kg
	Chrysene	24 µg/kg
	Dibenzo(a,h)anthracene	24 µg/kg
	Dibenzofuran	24 µg/kg
	Fluoranthene	97 µg/kg
	Fluorene	24 µg/kg
	Indeno(1,2,3-cd)pyrene	24 µg/kg
	Naphthalene	24 µg/kg
	Phenanthrene	24 µg/kg
	Pyrene	97 µg/kg
	Benzene	1.4 µg/kg
	Ethylbenzene	1.4 µg/kg
	Naphthalene	1.4 µg/kg
	o-Xylene	1.4 µg/kg
J8K74 (08204458; WN01SD)	2-Methylnaphthalene	240 µg/kg
	Acenaphthene	24 µg/kg
	Acenaphthylene	240 µg/kg
	Anthracene	240 µg/kg
	Benzo(a)anthracene	240 µg/kg
	Benzo(a)pyrene	240 µg/kg
	Benzo(b)fluoranthene	240 µg/kg
	Benzo(g,h,i)perylene	240 µg/kg
	Benzo(k)fluoranthene	240 µg/kg
	Carbazole	24 µg/kg
	Chrysene	240 µg/kg
	Dibenzo(a,h)anthracene	240 µg/kg
	Dibenzofuran	24 µg/kg
	Fluoranthene	2,400 µg/kg
	Fluorene	240 µg/kg
	Indeno(1,2,3-cd)pyrene	240 µg/kg
	Naphthalene (SVOC)	240 µg/kg
	Phenanthrene	240 µg/kg
	Pyrene	2,400 µg/kg
	Benzene	1.3 µg/kg

	Ethylbenzene	1.3 µg/kg
	Naphthalene (VOC)	12 µg/kg
	o-Xylene	1.3 µg/kg
J8K75 (08204459; WN02SD)	2-Methylnaphthalene	25 µg/kg
	Acenaphthene	25 µg/kg
	Acenaphthylene	130 µg/kg
	Anthracene	130 µg/kg
	Benzo(a)anthracene	1,300 µg/kg
	Benzo(a)pyrene	1,300 µg/kg
	Benzo(b)fluoranthene	1,300 µg/kg
	Benzo(k)fluoranthene	1,300 µg/kg
	Benzo(g,h,i)perylene	1,300 µg/kg
	Carbazole	25 µg/kg
	Chrysene	1,300 µg/kg
	Dibenzo(a,h)anthracene	130 µg/kg
	Dibenzofuran	25 µg/kg
	Fluoranthene	1,300 µg/kg
	Fluorene	130 µg/kg
	Indeno(1,2,3-cd)pyrene	1,300 µg/kg
	Naphthalene	130 µg/kg
	Phenanthrene	1,300 µg/kg
	Pyrene	1,300 µg/kg
J8K76 (08204460; WN03SD)	Arsenic	1.4 mg/kg
	2-Methylnaphthalene	260 µg/kg
	Acenaphthene	97 µg/kg
	Acenaphthylene	260 µg/kg
	Anthracene	1,300 µg/kg
	Benzo(a)anthracene	1,300 µg/kg
	Benzo(a)pyrene	1,300 µg/kg
	Benzo(b)fluoranthene	1,300 µg/kg
	Benzo(g,h,i)perylene	260 µg/kg
	Benzo(k)fluoranthene	1,300 µg/kg
	Carbazole	26 µg/kg
	Chrysene	1,300 µg/kg
	Dibenzo(a,h)anthracene	260 µg/kg
	Dibenzofuran	26 µg/kg
	Fluoranthene	1,300 µg/kg
	Fluorene	260 µg/kg
	Indeno(1,2,3-cd)pyrene	260 µg/kg
	Naphthalene	260 µg/kg
	Phenanthrene	1,300 µg/kg
	Pyrene	1,300 µg/kg
J8K77 (08204460; WN04SD)	2-Methylnaphthalene	24 µg/kg
	Acenaphthene	24 µg/kg
	Acenaphthylene	240 µg/kg
	Anthracene	2,400 µg/kg
	Benzo(a)anthracene	2,400 µg/kg
	Benzo(a)pyrene	2,400 µg/kg
	Benzo(b)fluoranthene	240 µg/kg
	Benzo(g,h,i)perylene	240 µg/kg

	Benzo(k)fluoranthene	240 µg/kg
	Carbazole	24 µg/kg
	Chrysene	2,400 µg/kg
	Dibenzo(a,h)anthracene	240 µg/kg
	Dibenzofuran	24 µg/kg
	Fluoranthene	2,400 µg/kg
	Fluorene	240 µg/kg
	Indeno(1,2,3-cd)pyrene	240 µg/kg
	Naphthalene	24 µg/kg
	Phenanthrene	2,400 µg/kg
	Pyrene	2,400 µg/kg

CRQLs – Contract Required Quantitation Limits

µg/kg – micrograms per kilogram

mg/kg – milligrams per kilogram

SVOC – Semivolatile Organic Compound

VOC – Volatile Organic Compound

Re Bremerton CRQL SVOC Questions

From: Matheny.Don@epamail.epa.gov
Sent: Monday, March 28, 2011 7:44 AM
To: Woodke, Mark
Cc: Crawford.Jennifer@epamail.epa.gov; Wu.Raymond@epamail.epa.gov
Subject: Re: Bremerton CRQL SVOC Questions

Attachments: Narrative.pdf

Mark,

The lab was working with adjusted CRQLs as per the Modified Analysis (MA) request. Here is a copy of their case narrative.

For VOAs, calculate the sample adjusted CRQL using a CRQL of 1.0 (instead of 5) and for SVOCs use a CRQL of 68 (instead of 170).
This is to match against the lowest calibration standards that were used in order to meet the CRQLs for this MA.

Hopefully the information in the narrative will help.

(See attached file: Narrative.pdf)

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Contract No. EPW05032

Case No. 37435

SDG No. J8K92

SDG NARRATIVE

Mod 1568.0

SAMPLE RECEIPT:

On 06/06/08 @ 10:00 A.M. - Received one cooler via FedEx with shipment number 865614916017 and the cooler temperature was 2.8°C.

The cooler contained the following samples for VOA and BNA analyses.
The custody seals and the samples were intact.

EPA SAMPLE ID	pH	EPA SAMPLE ID	pH
J8K92	<2	J8K74DL	NA
J8K74	NA	J8K74MS	NA
J8K75	NA	J8K74MSD	NA
J8K76	NA	J8K75RE	
J8K77	NA	J8K76RE	
J8K78	NA	J8K77RE	
J8K75DL	NA	J8K74DL2	NA
J8K76DL	NA	J8K75DL2	
J8K77DL	NA	J8K76DL2	
J8K78DL	NA	J8K77DL2	

No problems were encountered during sample receiving and login.

TRACE VOLATILES:

All samples were analyzed on A-5973 GC/MS GC/MS using a 30 meters long RTX-VMS column having a 0.25mm ID and 3µm film thickness. The trap used was OV-1/Tenax/Silica Gel (Tekmar #6 CAT #14-1755-003). A 25 mL purge volume was used for all samples, blanks and standards. The concentrations of the standards and spikes were maintained at the levels required by the Statement of Work (SOW).


These samples were analyzed for Trace Volatiles as per SOM 1.2 statement of work and the per Modification 1568.0.

To meet the low CRQL's for MA1568.0 the instrument is calibrated at the following concentrations.

Level 1 – 0.25 ug/L

Level 2 – 0.50 ug/L

Level 3– 2.5 ug/L


9/15/08

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Contract No. EPW05032

Case No. 37435

SDG No. J8K92

SDG NARRATIVE

Mod 1568.0

Level 4 – 5.0 ug/L

Level 5 – 10 ug/L

The formula used to calculate the Sample concentration:

$$\text{Concentration in ug/L} = \frac{(A_x) (I_s) (DF)}{(A_{is}) (RRF) (V_o)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured.

A_{is} = Area of the characteristic ion (EICP) for the internal standard.

I_s = Amount of internal standard added in ng.

RRF = Mean relative Response Factor from the initial calibration standard.

V_o = Total Volume of water purged, in ml.

DF = Dilution Factor.

VOA SOIL SAMPLE:

The samples for Low-Med-VOA was analyzed on instrument B-5973 GC/MS using a 30 meters long RTX-VMS columns having a 0.25mm ID and 3µm film thickness. The trap used was OV-1/Tenax/Silica Gel (Tekmar #6 CAT #14-1755-003).

A 10 mL purge volume and heated purge was used for soil volatile sample analysis, blanks and calibration standards. The concentrations of the standards and spikes were maintained at the levels required by the Statement of Work (SOW).

These samples were analyzed for Trace Volatiles as per SOM 1.2 statement of work and per Modification 1568.0.

The sample J8K74 had high target compound concentration above the calibration range and was analyzed using the dilution. Both the analyses were reported and are billable.

No problems were encountered during the analysis of this sample.

The formula used to calculate the Sample concentration:

LOW-MED-VOA SOIL SAMPLE:

$$\text{Concentration in ug/L} = \frac{(A_x) (I_s) (DF)}{(A_{is}) (RRF) (W_s)(D)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured.


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Contract No. EPW05032

Case No. 37435

SDG No. J8K92

SDG NARRATIVE

Mod 1568.0

A_{is} = Area of the characteristic ion (EICP) for the internal standard.

I_s = Amount of internal standard added in ng.

RRF = Mean relative Response Factor from the initial calibration standard.

100 - % Moisture

$D = \frac{\text{100 - \% Moisture}}{100}$

100

W_s = Weight of sample added to the purge tube, in g.

To meet the low CRQL's for MA1568.0 the instrument is calibrated at the following concentrations.

Level 1 – 1.0 ug/ kg

Level 2 – 2.0 ug/ kg

Level 3– 10 ug/ kg

Level 4 – 20 ug/ kg

Level 5 – 40 ug/ kg

SEMIVOLATILES:

The soil sample was extracted on 05/28/08 using sonication method using 100 g as per statement of work SOM1.2. The sample was cleaned by the GPC. No problems were encountered during the extraction and analysis.

The samples were analyzed on instrument F-5973 GC/MS using a 30 meters long RTX-5MS column having a 0.25mm ID and 0.25µm film thickness.

The SVOA and SVSIM were analyzed as per Modification 1568.0.

The samples J8K75, J8K76 and J8K77 had failed in the internal standards and were reanalyzed. Upon reanalysis again failed due to sample matrix and both the analyses were reported and are billable.

The samples J8K74, J8K75, J8K76, J9K77 and J8K78 had high target compound concentrations and were analyzed using the dilutions. Both the analyses were reported and are billable.

The samples J8K74, J8K75, J8K76 and J9K77 were analyzed using multiple dilutions in order to bring the target compound concentrations with in the calibration range. All the analyses were reported and are billable.

In the SVSIM analyses the closing calibration had failed due to carry over from the samples.

To meet the low CRQL's for MA1568.0 the instrument is calibrated at the following concentrations.

Level 1 – 2.0 ug/mL

Level 2 – 4.0 ug/mL

Level 3– 8.0 ug/mL

Level 4 – 16 ug/mL

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Contract No. EPW05032

Case No. 37435

SDG No. J8K92

SDG NARRATIVE

Mod 1568.0

Level 5 – 32 ug/mL

The formula used to calculate the Sample concentration:

SOIL SAMPLES:

$$\text{Concentration of Soil, Sediment sample ug/kg} = \frac{(A_x)(I_s)(V_t)(DF)(GPC)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

Where,

A_x , I_s , V_{in} , V_{out} are given for water, above.

V_t = Volume of concentrated extract in uL.

V_i = Volume of extract injected.

GPC = GPC cleaning Factor.

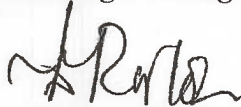
$$D = \frac{100 - \% \text{moisture}}{100}$$

W_s = Weight of sample extract.


RRF = Mean relative Response Factor determined from the initial calibration standard.

DF = Dilution Factor.

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hard copy sample data package and in the electronic data deliverable has been authorized by the laboratory manager or the manager's designee, as verified by the following signature:


Signature/Title

6/28/08
Date of Signature


6/15/08

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compound coelution, baseline noise, or matrix interferences. In these circumstances, the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific TCL compound. The area integrated shall not include baseline background noise. The area integrated shall not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet Quality Control (QC) criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instance of manual integration must be documented in the SDG Narrative.

- 11.2.1.3 In all instances where the data system report has been edited or where manual integration or quantitation has been performed, the GC/MS Operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "M" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards, and DMCs.
- 11.2.1.4 The requirements listed in Sections 11.2.1.1 - 11.2.1.3 apply to all standards, samples, and blanks.
- 11.2.1.5 The Mean Relative Response Factor (\overline{RRF}) from the initial calibration is used to calculate the concentration in the sample. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reason in the SDG Narrative. The area of a secondary ion cannot be used for the area of a primary ion unless a \overline{RRF} is calculated using the secondary ion.
- 11.2.1.6 Calculate the concentration in the sample using the \overline{RRF} and Equations 5 and 6.
- 11.2.1.6.1 Water

EQ. 5 Concentration of Water Sample

$$\text{Concentration } \mu\text{g/L} = \frac{(A_x) (I_s) (V_t) (DF) (GPC)}{(A_{is}) (\overline{RRF}) (V_o) (V_i)}$$

Where,

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

I_s = Amount of internal standard injected in ng.

V_o = Volume of water extracted in mL.

V_i = Volume of extract injected in μL .

Exhibit D Semivolatiles -- Section 11
Data Analysis and Calculations (Con't)

V_t = Volume of the concentrated extract in μL (If GPC Cleanup is performed, $V_t = V_{\text{out}}$).

$\overline{\text{RRF}}$ = Mean Relative Response Factor determined from the initial calibration standard.

$\text{GPC} = \frac{V_{\text{in}}}{V_{\text{out}}} = \text{GPC factor. (If no GPC is performed, GPC} = 1).$

V_{in} = Volume of extract loaded onto GPC column.

V_{out} = Volume of extract collected after GPC cleanup.

DF = Dilution Factor. The DF for analysis of water samples for semivolatiles by this method is defined as follows:

$$\text{DF} = \frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

If no dilution is performed, $\text{DF} = 1.0$.

11.2.1.6.2 Soil/Sediment

EQ. 6 Concentration of Soil/Sediment Sample

$$\text{Concentration } \mu\text{g/Kg (Dry weight basis)} = \frac{(A_x) (I_s) (V_t) (DF) (GPC)}{(A_{is}) (\overline{\text{RRF}}) (V_i) (W_s) (D)}$$

Where,

A_x , I_s , A_{is} , V_{in} , and V_{out} are as given for water, above.

V_t = Volume of the concentrated extract in μL
(If no GPC Cleanup is performed, then $V_t = 1000 \mu\text{L}$.
If GPC Cleanup is performed, then $V_t = V_{\text{out}}$).

V_i = Volume of the extract injected in μL .

$$D = \frac{100 - \% \text{ Moisture}}{100}$$

W_s = Weight of sample extracted in g.

$\text{GPC} = \frac{V_{\text{in}}}{V_{\text{out}}} = \text{GPC Factor}$

$\overline{\text{RRF}}$ = Mean Relative Response Factor determined from the initial calibration standard.

Exhibit D Semivolatiles -- Section 11
Data Analysis and Calculations (Con't)

DF = Dilution Factor. The DF for analysis of soil/sediment samples for semivolatiles by this method is defined as follows:

$$DF = \frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

A GPC factor of 2.0 is used to account for the amount of extract that is not recovered from the mandatory use of GPC cleanup. Concentrating the extract collected after GPC to 0.5 mL maintains the sensitivity of the soil/sediment method.

11.2.2 Non-Target Compound

An estimated concentration for non-target compounds tentatively identified shall be quantitated by the internal standard method. For quantitation, the nearest internal standard free of interferences shall be used. The equations for calculating concentration are the same as Equations 5 and 6. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compounds to be measured and the internal standard. An RRF of 1 is to be assumed. The resulting concentration shall be qualified as "J" (estimated, due to lack of a compound specific response factor), and "N" (presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target component. An estimated concentration should be calculated for all TICs as well as those identified as unknowns.

11.2.3 CRQL Calculations

11.2.3.1 Water Samples

EQ. 7 Aqueous Adjusted CRQL

$$\frac{\text{Adjusted}}{\text{CRQL}} = \frac{\text{Contract}}{\text{CRQL}} \times \frac{(V_x) (V_t) (DF)}{(V_o) (V_c)}$$

Where,

V_t , DF, and V_o are as given in Equation 5.

V_x = Contract sample volume (1000 mL).

V_c = Contract concentrated extract volume (1000 μL if GPC is not performed. If GPC was performed, then $V_c = V_{\text{out}}$).

Exhibit D Semivolatiles -- Section 11
Data Analysis and Calculations (Con't)

11.2.3.2 Soil/Sediment Samples

EQ. 8 Soil/Sediment Adjusted CRQL

$$\frac{\text{Adjusted}}{\text{CRQL}} = \frac{\text{Contract}}{\text{CRQL}} \times \frac{(W_x) (V_t) (DF)}{(W_s) (V_c) (D)}$$

Where,

V_t and DF = As given in Equation 5.

W_s and D = As given in Equation 6.

W_x = Contract sample weight (30 g for low-level soil/sediment samples and 1.0 g for medium-level soil/sediment samples).

V_c = Contract concentrated extract volume
(If GPC is required, $V_c = V_{out}$).

11.2.4 Deuterated Monitoring Compound (DMC) Recoveries

11.2.4.1 Calculate DMC recoveries for all samples, blanks, and Matrix Spike and Matrix Spike Duplicates (MS/MSDs). Determine if recovery is within limits (Table 6) and report on the appropriate form.

11.2.4.2 Calculate the concentrations of the DMCs using the same equations as used for the target compounds. Calculate the recovery of each DMC using the following equation:

EQ. 9 DMC Percent Recovery Calculation

$$\% \text{ Recovery} = \frac{(\text{Concentration (or amount) found} \times DF)}{\text{Concentration (or amount) spiked}} \times 100$$

Where,

DF = Same as EQ. 5.

11.3 Technical Acceptance Criteria for Sample Analysis

11.3.1 The samples must be analyzed on a GC/MS system meeting the instrument performance check, initial calibration, CCV, and blank technical acceptance criteria. The sample must undergo cleanup procedures, when required, on a GPC meeting the technical acceptance criteria for GPC calibration.

11.3.2 The sample must be extracted and analyzed within the contract holding times.

11.3.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

11.3.4 The Percent Recoveries of DMCs in a sample must be within the recovery limits listed in Table 6. Up to four DMCs per sample may fail to meet the recovery limits listed in Table 6 but all Percent

11.2.1.2 Water

EQ. 7 Water Concentration Calculation

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x) (I_s) (DF)}{(A_{is}) (\overline{\text{RRF}}) (V_o)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target compounds, internal standards, and DMCs are listed in Table 2.

A_{is} = Area of the characteristic ion (EICP) for the internal standard. The target compounds are listed with their associated internal standards in Table 3.

I_s = Amount of internal standard added, in ng.

$\overline{\text{RRF}}$ = Mean Relative Response Factor from the initial calibration.

V_o = Total volume of water purged, in mL.

DF = Dilution Factor. The DF for analysis of water samples for volatiles by this method is defined as the ratio of the number of mL of water purged (i.e., V_o above) to the number of mL of the original water sample used for purging. For example, if 2.0 mL of sample is diluted to 5.0 mL with reagent water and purged, $DF = 5.0 \text{ mL} / 2.0 \text{ mL} = 2.5$. If no dilution is performed, $DF = 1.0$.

11.2.1.3 Low-Level Soil/Sediment

EQ. 8 Low-Level Soil/Sediment Concentration Calculation

$$\text{Concentration } (\mu\text{g/Kg}) \text{ (dry weight basis)} = \frac{(A_x) (I_s) (DF)}{(A_{is}) (\overline{\text{RRF}}) (W_s) (D)}$$

Where,

A_x , I_s , A_{is} , and DF are as given for water, Equation 7.

$\overline{\text{RRF}}$ = Mean Relative Response Factor from the heated purge of the initial calibration.

$$D = \frac{100 - \% \text{Moisture}}{100}$$

W_s = Weight of sample added to the purge tube, in g.

11.2.1.4 Medium-Level Soil/Sediment

Exhibit D Low/Medium Volatiles -- Section 11
Data Analysis and Calculations (Con't)

EQ. 9 Medium-Level Soil/Sediment Concentration Calculation

$$\text{Concentration } \mu\text{g/Kg (dry weight basis)} = \frac{(A_x) (I_s) (AV_t) (1000) (DF)}{(A_{is}) (\overline{RRF}) (V_a) (W_s) (D)}$$

Where,

A_x , I_s , A_{is} are as given for water, Equation 7.

\overline{RRF} = Mean Relative Response Factor from the **ambient** temperature purge of the initial calibration.

AV_t = Adjusted total volume of the methanol extract plus soil water in milliliters (mL) determined by:

$$AV_t = V_t + \{W_s - [W_s(D)]\}$$

Where V_t = total volume of methanol extract in milliliters (mL). This volume is typically 10 mL, even though only 1.0 mL is transferred to the vial in Section 10.1.5.5. The quantity derived from $\{W_s - [W_s(D)]\}$ is the soil water volume and is expressed in mL.

V_a = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 μL), in microliters (μL) added to reagent water for purging.

W_s = Weight of soil/sediment extracted, in g.

$$D = \frac{100 - \% \text{Moisture}}{100}$$

DF = Dilution Factor. The DF for analysis of soil/sediment samples for volatiles by the medium-level method is defined as:

$$\frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

- 11.2.1.5 For water, low-level and medium-level soil/sediment samples, xylenes are to be reported as "m,p-xylenes" and "o-xylene". Because m- and p-xylene isomers coelute, special attention must be given to the quantitation of the xylenes. In quantitating sample concentrations, be sure to use the correct corresponding Relative Response Factor (RRF) values.

NOTE: The area of each peak (i.e., the peaks for o-xylene and m,p-xylene) must appear on the complete quantitation report.

- 11.2.1.6 The stereoisomers, trans-1,2-dichloroethene, and cis-1,2-dichloroethene are to be reported separately.
- 11.2.1.7 Secondary ion quantitation is allowed **only** when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative. A secondary ion cannot be used unless an RRF is calculated using the secondary ion.

Exhibit D Low/Medium Volatiles -- Section 11
Data Analysis and Calculations (Con't)

- 11.2.1.8 The requirements listed in Sections 11.2.1.9 and 11.2.1.10 apply to all standards, samples including Matrix Spikes and Matrix Spike Duplicates (MS/MSDs) and blanks.
- 11.2.1.9 It is expected that situations will arise where the automated quantitation procedures in the GC/MS software provide inappropriate quantitations. This normally occurs when there is compound coelution, baseline noise, or matrix interferences. In these circumstances, the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific target compound, DMC, or internal standard compound. The area integrated shall not include baseline background noise. The area integrated shall also not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet Quality Control (QC) criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instances of manual integration must be documented in the SDG Narrative.
- 11.2.1.10 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS Operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "M" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Low/Medium Volatiles), internal standards, and DMCs.
- 11.2.2 Non-Target Compounds
- 11.2.2.1 An estimated concentration for non-target TICs shall be determined by the internal standard method. For quantitation, the nearest internal standard free of interferences shall be used.
- 11.2.2.2 The formulas for calculating non-target compound concentrations are the same as in Sections 11.2.1.2, 11.2.1.3, and 11.2.1.4. Total area counts (or peak heights) from the total Reconstructed Ion Chromatograms (RICs) are to be used for both the non-target compound to be measured (A_x) and the internal standard (A_{is}). An RRF of 1.0 is to be assumed. The value from this quantitation shall be qualified as "J" (estimated due to the lack of a compound-specific RRF), and "N" (presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target compound. An estimated concentration must be calculated for all TICs, as well as those identified as unknowns.

Exhibit D Low/Medium Volatiles -- Section 11
Data Analysis and Calculations (Con't)

11.2.3 CRQL Calculations

11.2.3.1 Water

EQ. 10 Water Adjusted CRQL Calculation

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{V_x}{V_o} \times \text{DF}$$

Where,

Contract CRQL = Exact CRQL values in Exhibit C of the
Statement of Work (SOW).

V_o and DF are as given in Equation 7.

V_x = Contract Sample Volume (5.0 mL).

11.2.3.2 Low-Level Soil/Sediment

EQ. 11 Low-Level Soil Adjusted CRQL Calculation

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(W_x)}{(W_s)(D)}$$

Where,

W_s and D are as given in Equation 8.

W_x = Contract Sample Weight (5.0 g).

11.2.3.3 Medium-Level Soil/Sediment

EQ. 12 Medium-Level Soil/Sediment Adjusted CRQL Calculation

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(W_x)(V_t)(V_y)(1000)(\text{DF})}{(W_s)(V_c)(V_a)(D)}$$

Where,

V_t , DF, W_s , V_a and D are as given in Equation 9.

W_x = Contract Sample Weight (5.0 g).

V_y = Contract Soil Aliquot Volume from soil methanol
extract (100 μ L).

V_c = Contract Soil Methanol Extract Volume (5,000 μ L).

11.2.4 Deuterated Monitoring Compound (DMC) Recoveries



ecology and environment, inc.

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MEMORANDUM

DATE: March 28, 2011

TO: Renee Nordeen, Project Manager, Ecology and Environment, Inc., Seattle, WA

FROM: Mark Woodke, START QA Chemist, Ecology and Environment, Inc., Seattle, WA *MW*

SUBJ: TDD: 10-11-0007

Example adjusted Contract Required Quantitation Limit (CRQL) calculations for soil samples collected in conjunction with the Bremerton Gasworks Targeted Brownfields Assessment conducted by Ecology and Environment, Inc., in 2008 are provided below. Actual adjusted CRQLs are provided at the end of this memo.

SOIL SAMPLES

The detection limits are derived from the contract required quantitation limits (CRQLs; attached), % solids sample weights, and dilution factors (all on the Form I's), and contract modifications (attached).

CLP Lab Soil/Sediment Inorganics Example Adjusted CRQL Calculation - Method ILM05.4

Adjusted CRQL = Contract CRQL x 1/% solids

Cobalt in sample MJ8K76

Adjusted CRQL = 5.0 mg/kg x 1/0.722 = 6.9 mg/kg

CLP Lab Soil/Sediment Organics Formula

SVOCs

Adjusted CRQL = Contract CRQL x Contract Sample Weight (30 grams)/Actual Sample Weight x Volume of Concentrated Extract/Concentrated Extract Volume (same if GPC is used, so they cancel out) x Dilution Factor/(1/% solids (decimal))

Example Calculation for sample J8K78

Acenaphthene - Method SOM01.2 (SVOC)

The Contract CRQL was modified for this project from 170 µg/kg to 68 µg/kg

68 µg/kg x 30 grams/100.3 grams 1/0.82 = 24 µg/kg.

VOCs

Adjusted CRQL = Contract CRQL x Contract Sample Weight (5 grams)/Actual Sample Weight x 1/% solids (decimal)

Example Calculation for sample J8K74

o-Xylene - Method SOM01.2 (VOC)

The Contract CRQL was modified for this project from 5 µg/kg to 1 µg/kg

1 µg/kg x 5 grams/4.6 grams x 1/0.82 = 1.3 µg/kg.

Former Grit Storage Area Source Soil Samples SQLs

Sample Identification	Hazardous Substance	Sample Quantitation Limit
J8K78 (08204462; WN05SD)	Arsenic	1.2 mg/kg
	2-Methylnaphthalene	24 µg/kg
	Acenaphthene	24 µg/kg
	Acenaphthylene	24 µg/kg
	Anthracene	24 µg/kg
	Benzo(a)anthracene	24 µg/kg
	Benzo(a)pyrene	24 µg/kg
	Benzo(b)fluoranthene	24 µg/kg
	Benzo(g,h,i)perylene	24 µg/kg
	Benzo(k)fluoranthene	24 µg/kg
	Carbazole	24 µg/kg
	Chrysene	24 µg/kg
	Dibenzo(a,h)anthracene	24 µg/kg
	Dibenzofuran	24 µg/kg
	Fluoranthene	97 µg/kg
	Fluorene	24 µg/kg
	Indeno(1,2,3-cd)pyrene	24 µg/kg
	Naphthalene	24 µg/kg
	Phenanthrene	24 µg/kg
	Pyrene	97 µg/kg
	Benzene	1.4 µg/kg
	Ethylbenzene	1.4 µg/kg
	Naphthalene	1.4 µg/kg
	o-Xylene	1.4 µg/kg
J8K74 (08204458; WN01SD)	2-Methylnaphthalene	240 µg/kg
	Acenaphthene	24 µg/kg
	Acenaphthylene	240 µg/kg
	Anthracene	240 µg/kg
	Benzo(a)anthracene	240 µg/kg
	Benzo(a)pyrene	240 µg/kg
	Benzo(b)fluoranthene	240 µg/kg
	Benzo(g,h,i)perylene	240 µg/kg
	Benzo(k)fluoranthene	240 µg/kg
	Carbazole	24 µg/kg
	Chrysene	240 µg/kg
	Dibenzo(a,h)anthracene	240 µg/kg
	Dibenzofuran	24 µg/kg
	Fluoranthene	2,400 µg/kg
	Fluorene	240 µg/kg
	Indeno(1,2,3-cd)pyrene	240 µg/kg
	Naphthalene (SVOC)	240 µg/kg
	Phenanthrene	240 µg/kg
	Pyrene	2,400 µg/kg
	Benzene	1.3 µg/kg

	Ethylbenzene	1.3 µg/kg
	Naphthalene (VOC)	12 µg/kg
	o-Xylene	1.3 µg/kg
J8K75 (08204459; WN02SD)	2-Methylnaphthalene	25 µg/kg
	Acenaphthene	25 µg/kg
	Acenaphthylene	130 µg/kg
	Anthracene	130 µg/kg
	Benzo(a)anthracene	1,300 µg/kg
	Benzo(a)pyrene	1,300 µg/kg
	Benzo(b)fluoranthene	1,300 µg/kg
	Benzo(k)fluoranthene	1,300 µg/kg
	Benzo(g,h,i)perylene	1,300 µg/kg
	Carbazole	25 µg/kg
	Chrysene	1,300 µg/kg
	Dibenzo(a,h)anthracene	130 µg/kg
	Dibenzofuran	25 µg/kg
	Fluoranthene	1,300 µg/kg
	Fluorene	130 µg/kg
	Indeno(1,2,3-cd)pyrene	1,300 µg/kg
	Naphthalene	130 µg/kg
	Phenanthrene	1,300 µg/kg
	Pyrene	1,300 µg/kg
J8K76 (08204460; WN03SD)	Arsenic	1.4 mg/kg
	2-Methylnaphthalene	260 µg/kg
	Acenaphthene	97 µg/kg
	Acenaphthylene	260 µg/kg
	Anthracene	1,300 µg/kg
	Benzo(a)anthracene	1,300 µg/kg
	Benzo(a)pyrene	1,300 µg/kg
	Benzo(b)fluoranthene	1,300 µg/kg
	Benzo(g,h,i)perylene	260 µg/kg
	Benzo(k)fluoranthene	1,300 µg/kg
	Carbazole	26 µg/kg
	Chrysene	1,300 µg/kg
	Dibenzo(a,h)anthracene	260 µg/kg
	Dibenzofuran	26 µg/kg
	Fluoranthene	1,300 µg/kg
	Fluorene	260 µg/kg
	Indeno(1,2,3-cd)pyrene	260 µg/kg
	Naphthalene	260 µg/kg
	Phenanthrene	1,300 µg/kg
	Pyrene	1,300 µg/kg
J8K77 (08204460; WN04SD)	2-Methylnaphthalene	24 µg/kg
	Acenaphthene	24 µg/kg
	Acenaphthylene	240 µg/kg
	Anthracene	2,400 µg/kg
	Benzo(a)anthracene	2,400 µg/kg
	Benzo(a)pyrene	2,400 µg/kg
	Benzo(b)fluoranthene	240 µg/kg
	Benzo(g,h,i)perylene	240 µg/kg

	Benzo(k)fluoranthene	240 µg/kg
	Carbazole	24 µg/kg
	Chrysene	2,400 µg/kg
	Dibenzo(a,h)anthracene	240 µg/kg
	Dibenzofuran	24 µg/kg
	Fluoranthene	2,400 µg/kg
	Fluorene	240 µg/kg
	Indeno(1,2,3-cd)pyrene	240 µg/kg
	Naphthalene	24 µg/kg
	Phenanthrene	2,400 µg/kg
	Pyrene	2,400 µg/kg

µg/kg – micrograms per kilogram

mg/kg – milligrams per kilogram

SVOC – Semivolatile Organic Compound

VOC – Volatile Organic Compound



Multi-Media, Multi-Concentration, Inorganic Analytical Service for Superfund (ILM05.4)

Office of Superfund Remediation & Technology Innovation
Analytical Services Branch (5203P)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to EPA at such sites is a series of inorganic analytes and cyanide that are analyzed using Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES), Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), Cold Vapor Atomic Absorption (CVAA), and colorimetric techniques. The Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) offers an analytical service that provides data from the analysis of water/aqueous and soil/sediment samples for inorganic analytes for use in the Superfund and other decision making processes. Through a series of standardized procedures and a strict chain-of-custody, the inorganic analytical service produces data of known and documented quality. This service is available through the Superfund Contract Laboratory Program (CLP).

DESCRIPTION OF SERVICES

The inorganic analytical service provides a technical and contractual framework for laboratories to utilize EPA/CLP analytical methods. These methods are used in the isolation, detection and quantitative measurement of 23 target analyte metals (including mercury) and cyanide in both water and soil/sediment environmental samples. The CLP provides the methods to be used and the specific technical, reporting, and contractual requirements, including Quality Assurance (QA), Quality Control (QC), and Standard Operating Procedures (SOPs), by which EPA evaluates the data.

Three data delivery turnaround times are available to CLP customers: 7, 14, and 21-day turnaround after receipt of the last sample in the set. A 72-hour preliminary data submission option also is available for all turnaround times. The data associated with these Preliminary Results is due within 72 hours after receipt of each sample at the laboratory. In addition, data users may include, but are not limited to, additional analytes and modified quantitation limits.

The ILM05.4 analytical service is based on the previous ILM05.3 analytical service. The update to ILM05.4 includes a change to the Contract Required Quantitation Limits (CRQL) for the Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) analysis for vanadium in water matrices from 1 $\mu\text{g/L}$ to 5 $\mu\text{g/L}$.

DATA USES

This analytical service provides data that EPA uses for a variety of purposes. Examples include determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of a hazardous waste site including: site inspections, Hazard Ranking System scoring, remedial investigations/feasibility studies, remedial design, treatability studies, and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/litigation activities.

TARGET ANALYTES

The inorganic analytes and quantitation limits for which this service is applicable are listed in Table 1. Specific detection limits are method and matrix dependent.

The list of target analytes for this service was originally derived from the EPA Priority Pollutant List of 129 compounds. In the years since the inception of the CLP, analytes have been added to and deleted from the Target Analyte List (TAL), based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program.

Table 1. Inorganic Target Analyte List and Contract Required Quantitation Limits (CROLs)

<u>Analyte</u>	<u>ICP-AES CROL for Water (µg/L)</u>	<u>ICP-AES CROL for Soil (mg/kg)</u>	<u>ICP-MS CROL for Water (µg/L)</u>
1. Aluminum	200	20	--
2. Antimony	60	6	2
3. Arsenic	10	1	1
4. Barium	200	20	10
5. Beryllium	5	0.5	1
6. Cadmium	5	0.5	1
7. Calcium	5000	500	--
8. Chromium	10	1	2
9. Cobalt	50	5	1
10. Copper	25	2.5	2
11. Iron	100	10	--
12. Lead	10	1	1
13. Magnesium	5000	500	--
14. Manganese	15	1.5	1
15. Mercury	0.2	0.1	--
16. Nickel	40	4	1
17. Potassium	5000	500	--
18. Selenium	35	3.5	5
19. Silver	10	1	1
20. Sodium	5000	500	--
21. Thallium	25	2.5	1
22. Vanadium	50	5	5
23. Zinc	60	6	2
24. Cyanide	10	2.5	--

METHODS AND INSTRUMENTATION

The Contractor will demonstrate the ability to meet certain program data quality objectives prior to analyzing field samples. The laboratories must document methods used to generate analytical results and determine Method Detection Limits (MDLs). ICP-Atomic Emission Spectroscopy (ICP-AES) is used to analyze water, sediment, sludge, and soil samples. Water and soil samples are treated with acids and heated. The digestates are then analyzed for trace metals by an atomic emission optical spectroscopic technique. The samples are nebulized and the aerosol is transported to a plasma torch. The atomic-line emission spectra are dispersed and a photosensitive device monitors line intensities.

ICP-Mass Spectrometry (ICP-MS) is used to determine the concentration of dissolved and total recoverable elements in water/aqueous samples. The sample material is introduced, by nebulization, into radio frequency plasma where desolvation, atomization, and ionization take place. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated based on their mass-to-charge ratio.

Cold Vapor Atomic Absorption (CVAA) is used to analyze water, sediment, sludge, and soil samples for total mercury. Organo-mercury compounds may also be present and will need to be broken down and converted to mercuric ions to respond to the CVAA techniques. For water samples, organic compounds are oxidized and then

reacted with a strong reducing agent. The volatile free mercury is then driven from the reaction flask by bubbling air through the solution. The air stream carries the mercury atoms to an absorption cell, which is then placed in the light path of the AA spectrophotometer. For soil/sediment, the samples undergo acid digestion/oxidation followed by reduction and measurement by conventional cold vapor technique.

Various water types, sediment, sludge, and soil samples are also analyzed for total cyanide. Hydrocyanic acid (HCN) is released through a reflux-distillation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion is determined colorimetrically by converting it to cyanogen chloride (CNCl).

Table 2 summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for this service include both hardcopy/electronic data reporting forms and supporting raw data. The laboratory must submit data to EPA within 7-, 14- or 21-days, or preliminary data must be submitted within 72 hours after laboratory receipt of each sample in the set, if requested. EPA then processes the data through an automated Data Assessment Tool (DAT). DAT is a complete CLP data assessment package. DAT incorporates Contract Compliance Screening (CCS) and Computer-Aided Data Review and Evaluation (CADRE)

Table 2. Methods and Instruments

Analyte	Instrument	Method
Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn	Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES)	ICP analysis of atomic-line emission spectra.
Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, V, Zn	ICP - Mass Spectrometry (ICP-MS)	ICP analysis of ions separated on basis of mass-to-charge ratio.
Mercury (Hg)	Cold Vapor Atomic Absorption (CVAA)	Acid digestion/oxidation followed by reduction and CVAA analysis.
Cyanide (CN)	Colorimeter or Spectrophotometer	Distillation followed by colorimetric analysis.

Table 3. Quality Control

QC Operation	Frequency
Instrument Calibration	Daily or each time instrument is set up.
Initial Calibration Verification	Following each instrument calibration for each wavelength or mass used.
Initial Calibration Blank	Following each instrument calibration, immediately after the Initial Calibration Verification (ICV).
Continuing Calibration Verification	For each wavelength or mass used, at a frequency of 10% or every two hours of a run, whichever is more frequent, and at the beginning and end of each run.
Continuing Calibration Blank	10% or every two hours of a run, whichever is more frequent, and at the beginning and end of each run. Performed immediately after the last Continuing Calibration Verification (CCV).
CRQL Check Standard (CRI)	Every 20 analytical samples and at the beginning and end of each run, but not before the ICV. Performed before the Interference Check Sample.
Interference Check Sample	For ICP-AES, every 20 analytical samples and at the beginning and end of each run, immediately after the CRI. For ICP-MS, at the beginning of the run.
Serial Dilution for ICP	For each matrix type or for each SDG, whichever is more frequent.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples.
Laboratory Control Sample	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, except aqueous mercury and cyanide.
Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Post Digestion/Distillation Spike	Each time Spike Sample Recovery is outside QC limits.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
ICP-MS Tune	Prior to calibration.
Method Detection Limit Determination	Prior to contract, annually thereafter, and after major instrument maintenance.
Interelement Corrections	Prior to contract, quarterly thereafter, and after major instrument adjustment.
Linear Range Analysis	Prior to contract, and quarterly thereafter.

review to provide EPA Regions with electronic reports (PC-compatible reports, spreadsheets, and electronic files) within 24 to 48 hours from the receipt of the data. This automated tool facilitates the transfer of analytical data into Regional databases. DAT can also be used to assist in the data validation process at the Region. In addition to the Regional electronic reports, the CLP laboratories are provided with a data assessment report that documents the instances of noncompliance. The laboratory has four business days to reconcile defective data and resubmit the data to EPA. EPA then reviews the data for noncompliance and sends a final data assessment report to the CLP laboratory and the Region.

QUALITY ASSURANCE

The Quality Assurance (QA) process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity and ensures that the data provided are of the quality required. During the data collection effort, QA activities ensure that the Quality Control (QC) system is functioning effectively and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data obtained to determine its suitability to support enforcement or remedial decisions. Each contract laboratory will establish a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, functional guidelines, and specific QA/QC activities designed to achieve the data quality requirements for this analytical service.

QUALITY CONTROL

The QC process includes those activities required during analytical data collection to produce data of known and documented quality.

The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analytical service are shown in **Table 3**.

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by ASB and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, and evaluates laboratory performance with blind performance evaluation samples.

For more information, or for suggestions to improve this analytical service, please contact:

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Multi-Media, Multi-Concentration, Organic Analytical Service for Superfund (SOM01.2)

Office of Superfund Remediation and Technology Innovation (OSRTI)
Analytical Services Branch (ASB) (5203P)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to the EPA at such sites are a series of volatile, semivolatile, pesticide, and Aroclor compounds that are analyzed using gas chromatography coupled with mass spectrometry (GC/MS) and gas chromatography with an electron capture detector (GC/ECD). The Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) offers an analytical service that provides data from the analysis of water and soil/sediment samples for organic compounds for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the organic analytical service produces data of known and documented quality. This service is available through the Superfund Contract Laboratory Program (CLP).

DESCRIPTION OF SERVICES

This new organic analytical service provides a technical and contractual framework for laboratories to apply EPA/CLP analytical methods for the isolation, detection, and quantitative measurement of 52 volatile, 67 semivolatile, 21 pesticide, and 9 Aroclor target compounds in water and soil/sediment environmental samples. The CLP provides the methods to be used and the specific technical, reporting, and contractual requirements, including Quality Assurance (QA), Quality Control (QC), and Standard Operating Procedures (SOPs), by which EPA evaluates the data. This service uses GC/MS and GC/ECD methods to analyze the target compounds.

Three data delivery turnarounds are available to CLP customers: 7-day, 14-day, and 21-day turnaround after laboratory receipt of the last sample in the set. In addition, there are 48-hour (for trace volatiles and volatiles) and 72-hour (for semivolatiles, pesticides, and Aroclors) preliminary data submission options available. Options under this service include a closed system purge-and-trap method for low-level volatile soil analysis and methanol preservation for medium-level volatile soil analysis. In addition, data users may request modifications to the SOW that may include, but are not limited to, additional compounds, sample matrices other than soil/sediment or water, lower quantitation limits, and other requirements to enhance method performance.

DATA USES

This analytical service provides data which EPA uses for a variety of purposes, such as determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of a hazardous waste site including, but not limited to: site inspections; Hazard Ranking System (HRS) scoring; remedial investigations/Feasibility Studies (FSs); remedial design; treatability studies; and removal actions. In addition, this service provides data that will be available for use in Superfund enforcement/litigation activities.

TARGET COMPOUNDS

Table 1 lists the compounds for which this service is applicable and the corresponding quantitation limits. Specific quantitation limits are highly matrix-dependent.

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) for SOM01.2*

Quantitation Limits						Quantitation Limits					
	Trace Water by SIM (µg/L)	Trace Water (µg/L)	Low Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)		Trace Water by SIM (µg/L)	Trace Water (µg/L)	Low Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)
<u>VOLATILES</u>						<u>VOLATILES (CON'T)</u>					
1. Dichlorodifluoromethane		0.50	5.0	5.0	250	40. Ethylbenzene		0.50	5.0	5.0	250
2. Chloromethane		0.50	5.0	5.0	250	41. o-Xylene		0.50	5.0	5.0	250
3. Vinyl Chloride		0.50	5.0	5.0	250	42. m, p-Xylene		0.50	5.0	5.0	250
4. Bromomethane		0.50	5.0	5.0	250	43. Styrene		0.50	5.0	5.0	250
5. Chloroethane		0.50	5.0	5.0	250	44. Bromoform		0.50	5.0	5.0	250
6. Trichlorofluoromethane		0.50	5.0	5.0	250	45. Isopropylbenzene		0.50	5.0	5.0	250
7. 1,1-Dichloroethene		0.50	5.0	5.0	250	46. 1,1,2,2-Tetrachloroethane		0.50	5.0	5.0	250
8. 1,1,2-Trichloro-1,2,2-trifluoroethane		0.50	5.0	5.0	250	47. 1,3-Dichlorobenzene		0.50	5.0	5.0	250
9. Acetone		5.0	10	10	500	48. 1,4-Dichlorobenzene		0.50	5.0	5.0	250
10. Carbon Disulfide		0.50	5.0	5.0	250	49. 1,2-Dichlorobenzene		0.50	5.0	5.0	250
11. Methyl acetate		0.50	5.0	5.0	250	50. 1,2-Dibromo-3-chloropropane	0.050	0.50	5.0	5.0	250
12. Methylene chloride		0.50	5.0	5.0	250	51. 1,2,4-Trichlorobenzene		0.50	5.0	5.0	250
13. trans-1,2-Dichloroethene		0.50	5.0	5.0	250	52. 1,2,3-Trichlorobenzene		0.50	5.0	5.0	250
14. Methyl tert-butyl ether		0.50	5.0	5.0	250						
							Low Water by SIM (µg/L)	Low Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)
						<u>SEMIVOLATILES</u>					
15. 1,1-Dichloroethane		0.50	5.0	5.0	250	53. Benzaldehyde		5.0		170	5000
16. cis-1,2-Dichloroethene		0.50	5.0	5.0	250	54. Phenol		5.0		170	5000
17. 2-Butanone		5.0	10	10	500	55. bis-(2-chloroethyl) ether		5.0		170	5000
18. Bromochloromethane		0.50	5.0	5.0	250	56. 2-Chlorophenol		5.0		170	5000
19. Chloroform		0.50	5.0	5.0	250	57. 2-Methylphenol		5.0		170	5000
20. 1,1,1-Trichloroethane		0.50	5.0	5.0	250	58. 2,2'-Oxybis (1-chloropropane)		5.0		170	5000
21. Cyclohexane		0.50	5.0	5.0	250	59. Acetophenone		5.0		170	5000
22. Carbon tetrachloride		0.50	5.0	5.0	250	60. 4-Methylphenol		5.0		170	5000
23. Benzene		0.50	5.0	5.0	250	61. N-Nitroso-di-n propylamine		5.0		170	5000
24. 1,2-Dichloroethane		0.50	5.0	5.0	250	62. Hexachloroethane		5.0		170	5000
25. 1,4-Dioxane			100	100	5000	63. Nitrobenzene		5.0		170	5000
26. Trichloroethene		0.50	5.0	5.0	250	64. Isophorone		5.0		170	5000
27. Methylcyclohexane		0.50	5.0	5.0	250	65. 2-Nitrophenol		5.0		170	5000
28. 1,2-Dichloropropane		0.50	5.0	5.0	250	66. 2,4-Dimethylphenol		5.0		170	5000
29. Bromodichloromethane		0.50	5.0	5.0	250	67. Bis (2-chloroethoxy) methane		5.0		170	5000
30. cis-1,3-Dichloropropene		0.50	5.0	5.0	250	68. 2,4-Dichlorophenol		5.0		170	5000
31. 4-Methyl-2-pentanone		5.0	10	10	500	69. Napthalene	0.10	5.0	3.3	170	5000
32. Toluene		0.50	5.0	5.0	250	70. 4-Chloroaniline		5.0		170	5000
33. trans-1,3-Dichloropropene		0.50	5.0	5.0	250	71. Hexachlorobutadiene		5.0		170	5000
34. 1,1,2-Trichloroethane		0.50	5.0	5.0	250	72. Caprolactam		5.0		170	5000
35. Tetrachloroethane		0.50	5.0	5.0	250	73. 4-Chloro-3-methylphenol		5.0		170	5000
36. 2-Hexanone		5.0	10	10	500	74. 2-Methylnapthalene	0.10	5.0	3.3	170	5000
37. Dibromochloromethane		0.50	5.0	5.0	250	75. Hexachlorocyclo-pentadiene		5.0		170	5000
38. 1,2-Dibromochloroethane	0.050	0.50	5.0	5.0	250	76. 2,4,6-Trichlorophenol		5.0		170	5000
39. Chlorobenzene		0.50	5.0	5.0	250	77. 2,4,5-Trichlorophenol		5.0		170	5000

* For volatiles, quantitation limits for medium soils are approximately 50 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 30 times the quantitation limits for low soils.

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) for SOM01.2* (Con't)

Quantitation Limits						Quantitation Limits					
	Low Water by SIM (µg/L)	Low Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)		Low Water by SIM (µg/L)	Low Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)
SEMIVOLATILES (CON'T)						SEMIVOLATILES (CON'T)					
78. 1,1'-Biphenyl		5.0		170	5000	115. Benzo (a) pyrene	0.10	5.0	3.3	170	5000
79. 2-Chloronaphthalene		5.0		170	5000	116. Indeno (1,2,3-cd)-pyrene	0.10	5.0	3.3	170	5000
80. 2-Nitroaniline		10		330	10000	117. Dibenzo (a,h)-anthracene	0.10	5.0	3.3	170	5000
81. Dimethylphthalate		5.0		170	5000	118. Benzo (g,h,i) perylene	0.10	5.0	3.3	170	5000
82. 2,6-Dinitrotoluene		5.0		170	5000	119. 2,3,4,6-Tetrachlorophenol		5.0		170	5000
83. Acenaphthylene	0.10	5.0	3.3	170	5000	PESTICIDES					
84. 3-Nitroaniline		10		330	10000	Water (µg/L)			Soil (µg/kg)		
85. Acenaphthene	0.10	5.0	3.3	170	5000	120. alpha-BHC	0.050			1.7	
86. 2,4-Dinitrophenol		10		330	10000	121. beta-BHC	0.050			1.7	
87. 4-Nitrophenol		10		330	10000	122. delta-BHC	0.050			1.7	
88. Dibenzofuran		5.0		170	5000	123. gamma-BHC (Lindane)	0.050			1.7	
89. 2,4-Dinitrotoluene		5.0		170	5000	124. Heptachlor	0.050			1.7	
90. Diethylphthalate		5.0		170	5000	125. Aldrin	0.050			1.7	
91. Fluorene	0.10	5.0	3.3	170	5000	126. Heptachlor epoxide	0.050			1.7	
92. 4-Chlorophenyl-phenyl ether		5.0		170	5000	127. Endosulfan I	0.050			1.7	
93. 4-Nitroaniline		10		330	10000	128. Dieldrin	0.10			3.3	
94. 4,6-Dinitro-2-methylphenol		10		330	10000	129. 4,4'-DDE	0.10			3.3	
95. N-Nitrosodiphenylamine		5.0		170	5000	130. Endrin	0.10			3.3	
96. 1,2,4,5-Tetra chlorobenzene		5.0		170	5000	131. Endosulfan II	0.10			3.3	
97. 4-Bromophenyl-phenylether		5.0		170	5000	132. 4,4'-DDD	0.10			3.3	
98. Hexachlorobenzene		5.0		170	5000	133. Endosulfan sulfate	0.10			3.3	
99. Atrazine		5.0		170	5000	134. 4,4'-DDT	0.10			3.3	
100. Pentachlorophenol	0.20	10	6.7	330	10000	135. Methoxychlor	0.50			17	
101. Phenanthrene	0.10	5.0	3.3	170	5000	136. Endrin ketone	0.10			3.3	
102. Anthracene	0.10	5.0	3.3	170	5000	137. Endrin aldehyde	0.10			3.3	
103. Carbazole		5.0		170	5000	138. alpha-Chlordane	0.050			1.7	
104. Di-n-butylphthalate		5.0		170	5000	139. gamma-Chlordane	0.050			1.7	
105. Fluoranthene	0.10	5.0	3.3	170	5000	140. Toxaphene	5.0			170	
106. Pyrene	0.10	5.0	3.3	170	5000	AROCLORS			Water (µg/L)		
107. Butylbenzylphthalate		5.0		170	5000				Soil (µg/kg)		
108. 3,3'-Dichlorobenzidine		5.0		170	5000	141. Aroclor-1016	1.0			33	
109. Benzo (a) anthracene	0.10	5.0	3.3	170	5000	142. Aroclor-1221	1.0			33	
110. Chrysene	0.10	5.0	3.3	170	5000	143. Aroclor-1232	1.0			33	
111. Bis (2-ethylhexyl) phthalate		5.0		170	5000	144. Aroclor-1242	1.0			33	
112. Di-n-octylphthalate		5.0		170	5000	145. Aroclor-1248	1.0			33	
113. Benzo (b) fluoroanthene	0.10	5.0	3.3	170	5000	146. Aroclor-1254	1.0			33	
114. Benzo (k) fluoroanthene	0.10	5.0	3.3	170	5000	147. Aroclor-1260	1.0			33	
						148. Aroclor-1262	1.0			33	
						149. Aroclor-1268	1.0			33	

* For volatiles, quantitation limits for medium soils are approximately 50 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 30 times the quantitation limits for low soils.

The TCL for this service was originally derived from the EPA Priority Pollutant List of 129 compounds. In the years since the inception of the CLP, compounds have been added to and removed from the TCL, based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program. The SOM analytical service combines the previous OLM and OLC services into one method. For example, drinking water and ground water type samples may be analyzed using the Trace Volatiles method in SOM.

METHODS AND INSTRUMENTATION

For trace volatile water samples, 25 mL of water sample is added to a purge-and-trap device and purged with an inert gas at room temperature. For low/medium volatile water samples, 5 mL of water sample is added to a purge-and-trap device and purged with an inert gas at room temperature. Higher purge temperatures may be used for both trace and low/medium volatile analyses if all technical acceptance criteria is met for all standards, samples, and blanks. For low-level volatile soil samples, organic compounds are generally determined by analyzing approximately 5 g of sample in a closed-system purge-and-trap device at 40°C. For a medium-level soil sample, a soil sample of 5 g is collected, preserved, and/or extracted with methanol and an aliquot of methanol extract is added to 5 mL reagent water and purged at room temperature. For water and soil samples, the volatiles purged from the sample are trapped on a solid sorbent. The purged volatiles are subsequently desorbed by rapidly heating and backflushing with helium, and then introduced into a GC/MS system.

For semivolatile, pesticide, and Aroclor water samples, a 1 L aliquot of sample is extracted with methylene chloride using a continuous liquid-liquid extractor or separatory funnel (for pesticides and Aroclors only). For low-level semivolatile, pesticide, and Aroclor soil samples, a 30 g soil/sediment sample is extracted with methylene chloride/acetone using sonication, automated Soxhlet/Dean-Stark (SDS) extraction, or pressurized fluid extraction techniques. For medium-level semivolatile soil samples, a 1g aliquot is extracted with methylene chloride using the techniques mentioned above for low-level soil samples. For both water and soil samples, the extract is concentrated, subjected to fraction-specific cleanup procedures, and analyzed by GC/MS for semivolatiles or GC/ECD for pesticides and Aroclors. Table 2 summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for this service include hardcopy data reporting forms and supporting raw data. In addition to the hardcopy deliverable, contract laboratories must also submit the same data electronically. The laboratory must submit data to EPA within 7, 14, or 21-days after laboratory receipt of the last sample in set [or

preliminary data within 48 hours (for trace volatiles and volatiles) or 72 hours (for semivolatiles, pesticides, and Aroclors)] after laboratory receipt of each sample. EPA then processes the data through an automated Data Assessment Tool (DAT). DAT provides EPA Regions with PC-compatible reports, spreadsheets, and electronic files within 24-48 hours from the receipt of the data for use in data validation. This automated tool also facilitates the transfer of analytical data into Regional databases. In addition to the Regional electronic reports, the CLP laboratories are provided with a data assessment report that documents the instances of noncompliance. The laboratory has 6 business days to reconcile defective data and resubmit the data to EPA. EPA then reviews the data for noncompliance and sends a final data assessment report to the CLP laboratory and the Region.

QUALITY ASSURANCE (QA)

The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity. This process ensures that the data provided are of known and documented quality.

During the implementation of the data collection effort, QA activities ensure that the Quality Control (QC) system is functioning effectively and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data to determine its suitability to support enforcement or remedial decisions.

Each contract laboratory prepares a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, and functional guidelines, as well as the QA and QC activities designed to achieve the data quality requirements in the contract.

QUALITY CONTROL (QC)

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analytical service are provided in Table 3.

Table 2. Methods and Instruments

Fraction	Water	Soil
Trace Volatiles	Purge-and-trap followed by GC/MS analysis	N/A
Volatiles	Purge-and-trap followed by GC/MS analysis	Purge-and-trap or closed-system purge-and-trap followed by GC/MS analysis
Semivolatiles	Continuous liquid-liquid extraction (CLLE) followed by GC/MS analysis	Sonication, automated SDS extraction, or pressurized fluid extraction followed by GC/MS analysis
Pesticides	CLLE or separatory funnel extraction followed by dual column GC/ECD analysis	Sonication, automated SDS extraction or pressurized fluid extraction followed by dual column GC/ECD analysis
Aroclors	CLLE or separatory funnel extraction followed by dual column GC/ECD analysis	Sonication, automated SDS extraction or pressurized fluid extraction followed by dual column GC/ECD analysis

Table 3. Quality Control (QC)

QC Operation	Frequency
Deuterated Monitoring Compounds (DMCs) (trace volatiles, volatiles, and semivolatiles)	Added to each sample, standard, and blank
Surrogates (pesticides and Aroclors)	Added to each sample, standard, and blank
Method Blanks (trace volatiles and volatiles)	Analyzed at least every 12 hours for each matrix and level
Method Blanks (semivolatiles, pesticides, and Aroclors)	Prepared with each group of 20 samples or less of same matrix and level, or each time samples are extracted by the same procedure
Instrument Blank (trace volatiles and volatiles)	Analyzed after a sample which contains compounds at concentrations greater than the calibration range
Instrument Blank (pesticides and Aroclors)	Every 12 hours on each GC column used for analysis
Storage Blanks (trace volatiles and volatiles)	Prepared and stored with each set of samples
GC/MS Mass Calibration and Ion Abundance Patterns (trace volatiles, volatiles, and semivolatiles)	Every 12 hours for each instrument used for analysis
GC Resolution Check (pesticides)	Prior to initial calibration, on each instrument used for analysis
Initial Calibration	Upon initial set up of each instrument, and each time continuing calibration fails to meet the acceptance criteria
Continuing Calibration	Every 12 hours for each instrument used for analysis
Internal Standards (trace volatiles, volatiles, and semivolatiles)	Added to each sample, standard, and blank
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	Once every 20 or fewer samples of same fraction, matrix, and level in a Sample Delivery Group (SDG)
Laboratory Control Samples (LCSs) (pesticides and Aroclors)	Once every 20 or fewer samples of same fraction, matrix, and level in an SDG
Method Detection Limit (MDL)	Determined annually, per matrix and level

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by ASB and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, GC/MS and/or GC/ECD tape audits, and evaluates laboratory performance through the use of blind Performance Evaluation (PE) samples.

CONTACTING EPA

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